



Fabrication of hollow microspheres filled fly ash based foam geopolymers with ultra-low thermal conductivity and relative high strength

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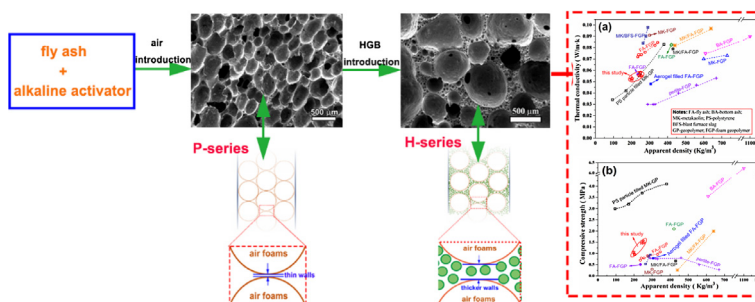
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HIGHLIGHTS

- A new framework of hollow microspheres filled foam geopolymer composites were developed.
- An easy and feasible method was developed to test air pore parameters (porosity, pore diameter, and through-hole rate).
- The materials fabricated in this study exhibit superior performance than most available geopolymer based materials to now.

GRAPHICAL ABSTRACT



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ABSTRACT

Fabrication of lightweight inorganic thermal insulation materials with high strength and ultra-low thermal conductivity is a great challenge and is receiving growing attention worldwide. Herein, a kind of geopolymer based materials with excellent performance were successfully fabricated by simultaneous incorporation of air foams and hollow microspheres in geopolymer matrix. Mechanical strength, bulk density, and thermal conductivity, were conducted as the key performance indexes for the evaluation of prepared samples in this study. On the other hand, materials foam structures, in terms of porosity, pore diameter, and through-hole rate, were analyzed by means of “image-fitting” method in this work. Compared to pure foamed geopolymer specimen (P-series), hollow microspheres filled foam geopolymers (H-series) behaved much better performance in strength and thermal insulation properties in same density levels. The optimal performances in terms of density versus compressive strength and thermal conductivity are as follows: (300 kg/m³ vs. 2.84 MPa vs. 0.0711 W/(m·K)), (250 kg/m³ vs. 1.57 MPa vs. 0.05509 W/(m·K)), and (200 kg/m³ vs. 1.06 MPa vs. 0.05223 W/(m·K)). The incorporation of hollow microspheres lead to the much thicker foam walls of H-series sample than P-series, which is regarded as the key reason of its highly improved performance.

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1. Introduction

Crises of energies, resources, and environment have globally broken out since the 21st century. As a result, environment protec-

tion, energy-saving, emission reduction, waste recycling, and new energy development, etc. have gradually become global consensus for the sustainable development of our living planet. Building energy conservation is one of the most important parts of energy-saving and emission reduction, and the most effective way to improve building energy efficiency is to use thermal insulation materials, based on which passive house can be took for example.

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Thermal insulation materials can be classified into three types: organics, inorganics, and organic-inorganic hybrids. Conventional inorganic thermal insulation materials, such as rock wool, glass wool, mineral wool, and expanded perlites, are the people initially used materials for building thermal insulation. However, mainly due to the defects of high energy and resources consumption during manufacturing processes [1,2], these conventional inorganic materials are gradually eliminated. Instead, organic types have become the predominant thermal insulation materials nowadays, which mainly includes expanded polystyrene, extruded polystyrene, polystyrene foam, polyurethane, etc. Compared with inorganic types, organic thermal insulation materials usually possess much lower thermal conductivity coefficient ($0.02\text{--}0.04\text{ W}/(\text{m}\cdot\text{K})$), ultra-low density ($20\text{--}100\text{ Kg}/\text{m}^3$), excellent ductility and water proofing properties [1,3]. Even so, due to the native attributes that belong to organics, these materials are usually easily shrinkable, labile, and flammable once they were heated or fired [4], which have caused severe damages to human lives and society wealth. Although some organic materials have achieved the “A-class” fire resistance by various modification methods [5,6], the negatively thermolabile behavior cannot be easily changed or they even produced toxic gases when the organics were heated, which have become the fatal defects. Similarly, organic-inorganic hybrid materials are also limited by the degraded organic nature. As a consequence, high-performance inorganic materials that simultaneously possess low thermo conductivity, A-class fire resistance, cost-effective, and high strength properties, have anew received growing attentions for building thermo insulation applications.

Geopolymers (GPs), also known as alkaline activated materials, are a class of inorganic polymer binder materials with Si—O—Si and Si—O—Al bonds in a highly cross linked X-ray amorphous network [7,8]. GPs usually show excellent thermal stability, relative high strength, low shrinkage, excellent fire resistance, and long service life [9,10]. Most of important, the manufacturing of GPs leave much smaller CO₂ footprint and consume much less energy than traditional cement, as they are usually obtained by chemically activation of industrial solid wastes (fly ash, metakaolin, slag, etc.) in mild conditions [9]. Based on its outstanding properties, GPs are regarded as one of the optimal alternatives for traditional cement. What's more, GPs also exhibit great potential in thermo insulation field, and great efforts have been paid on it. However, after making an overall survey on GPs based materials with excellent thermal insulation properties by us, most of them behaved high thermal conductivities ($>0.065\text{ W}/(\text{m}\cdot\text{K})$), high bulk densities ($>300\text{ Kg}/\text{m}^3$), and low strength [11–22]. Compared to the thermal insulation materials of organics [1,3], the GPs based materials to date possess weak competitiveness. There is no doubt that the thermo conductivity coefficient usually decreases as the apparent density decreased, but the mechanical strength also declines at the same time. The rare two available works reported by Duan et al. [11] and Vaou and Panias [17] obtained excellent thermo conductivities (as low as $0.03\text{--}0.04\text{ W}/(\text{m}\cdot\text{K})$), which can be comparable to organic thermo insulation materials. However, the study by Duan was on the polystyrene (PS) particle filled metakaolin based GPs, its ultra-high volume fraction of organic PS pose a potential threaten once applied for thermo insulation. Vaou otherwise reported perlite based GPs, but the relative high density and low strength faded the materials.

Based on the analysis of available works to date, this paper aims to fabricate a kind of wholly inorganic GP based materials with low thermal conductivity, low density, and relative high strength compared with like products. With this aim, a kind of fly ash based foam geopolymer composites filled with inorganic hollow microspheres were fabricated in this study. Apparent density, strength, thermo conductivity, air pores characteristics (including porosity, pore diameter, and corresponding through-hole rates) were

characterized and assessed in this paper. Hollow microspheres filled foam GPs fabricated in this study showed excellent properties in density, mechanical strength, and thermal conductivity when compared with available works to date. With its high performance and low CO₂ footprint, this new framework of GP composites may promote the development of inorganic materials for thermal insulation application significantly.

2. Experimental

2.1. Materials

Circulating fluidized combustion fly ash (CFA) supplied by Pingshuo coal gangue power plant (China Coal Group corp., Shanxi province) was employed in this study as the aluminosilicate resources (more details could be read from the author's previous study [23]). Hollow microspheres used in this study were the S38HS type hollow glass bubbles (HGB) from 3M Company, its typical properties and morphology were shown in Table 1 and Fig. 1, respectively. Alkaline activator (AA) used in this study was the hybrids of water glass (molar ratio SiO₂/Na₂O = 2.42, ~62 wt.% water content) and 8 M sodium hydroxide solutions in mass ratio of 1.23:1 to make the net concentration of [OH⁻] is ~4 M [24]. H₂O₂ (30 wt.%) was employed as the foaming agent, whereas calcium stearate (>99%, analytical pure) was used to be the foam stabilizer to control air foams structures and dimensions.

2.2. Specimen fabrication

Two different series of CFA based foam GPs were fabricated for contrastive study, one series is pure foamed GPs (denoted as P-series, ex. PA-1), and another series is foamed GPs filled with HGB (denoted as H-series, ex. HA-1). As the light-weight filler, the HGB dosage was designed to be fixed 30% (by weight of the total mass of CFA and HGB) for all H-series samples in this study. The dosage of H₂O₂ and calcium stearate were set as the variables to control porosity and pore structure of foam GPs, respectively. Detailed mix proportions were shown in Table 2, which were the experienced mix design after our repeated trials.

In accordance with the mix proportions in Table 2, CFA, HGB, and calcium stearate were firstly mixed and stirred to make homogeneously dry mixer. Then, under slow stirring, WG and NH(aq) were successively added into the dry mixer to make uniform slurry. After that, certain amount of H₂O₂ was added into the slurry and stirred in fast speed for 30 s. The obtained fresh paste was then cast into certain molds and coated with polyethylene thin films to hinder moisture in or out. At last, transferred the molds into a concrete standard curing box for 24 h curing (with curing temperature set as 60 °C). After curing procedure, the specimen were demoulded and exposed to ambient environment for free moisture releasing and further ambient curing. Based on different test requirements, two different dimen-

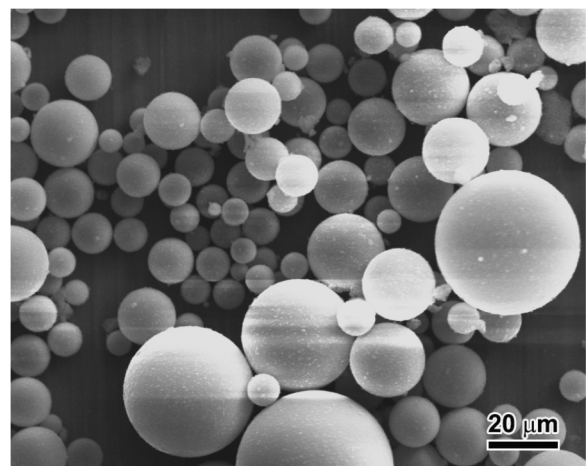


Fig. 1. SEM of HGB.

Table 1
Typical properties of S38HS type HGB used in this study.

	Apparent density/ Kg/m^3	Isostatic crush strength/ MPa	Thermal conductivity ($25\text{ }^\circ\text{C}$)/ $\text{W}/(\text{m}\cdot\text{K})$
HGB	150–180	37.9	0.127

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